Case Docket No. CHR 99-9

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Patent Application for

IMPROVEMENTS IN PAPERBOARD.

BACKGROUND OF THE INVENTION

1. <u>Field of the Invention</u>:

The present invention relates to a novel paperboard and method of producing paperboard, which contains an adsorptive material to effectively address the odor emission problem associated with such board. More particularly, the invention relates to a method of applying such adsorptive material in unbleached board in a way that does not negatively impact the appearance or physical attributes of the board.

2. <u>Description of Related Art (Including Information Disclosed Under 37 CFR 1.97 and 37 CFR 1.98)</u>

Various paperboard-based structures are utilized to store and/or serve liquid or solid, food or non-food, products. The odor issue related to paperboard, however, could have a negative impact for uses that are sensitive to inherent board odor, resulting from pulping chemicals, by-products, and processing additives. Various approaches have been utilized or reported to address the odor issue related to paperboard in general, in these structures. Some of these approaches are designed to combat odors generated by the material being packaged, rather than the odor of the board itself.

A widely known and used approach is to coat the paper-based structure with various barrier and sealant materials. One basic structure utilizes a three-layer laminate wall structure.

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The laminate comprises of a paperboard substrate coated on both sides by a layer of low-density polyethylene. A second widely known structure uses a five-layer laminate wall structure. This structure is comprised of paperboard substrate, a layer of low density polyethylene coated onto the foil layer rendering the structure heat sealable. In addition, various other barrier materials have been used to combat the transfer of various gases, light, and flavors into and out of the container. These approaches are for specific packages and add substantial cost to the package. Other approaches to address board odor involve using odor masking agents and adsorbents.

One common adsorbent is activated carbon. Adsorptive characteristics of activated carbon are well known. Carbon has been proposed to be used as blend in polyethylene where it could be coated onto the board to adsorb odors. Vinegar/carbon blends also have been suggested. These "coating" approaches with carbon might work, but they negatively impact the appearance of the board. Also, as the carbon is black, the impact on aesthetics of the board is highly undesirable.

Specific U.S. patents describing some of the above-discussed materials and methods include:

U.S. Patent No.	Title
4,212,852	"Method of Deodorizing Gas Containing Hydrogen Sulfide And Ammonia And/Or Amines"
4,235,027	"Laminated Insole"
4,256,728	"Deodorization Method"
4,337,276	"Method for Storing Produce and Container and Freshness Keeping Agent Therefor"

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U.S. Patent No. (cont'd)	Title
4,443,482	"Buttered Table Syrup in Polyolefin Bottle"
4,517,308	"Method of Producing a Sorptive Body, Particularly for Eliminating Odors, Air Freshening, Etc. and The Resultant Product"
4,528,281	"Carbon Molecular Sieves and a Process for Their Preparation and Use"
4,818,524	"Deodorizing Compositions"
4,840,823	"Plastic Film Packaging Material"
4,919,925	"Deodorant, Deodorizing Composite Material, Deodorizing Resin Composition, Deodorizing Resin Articles and Deodorizing Foam"
4,931,360	"Deodorizing Sheet with a Deodorizing Coating Formulation"
4,938,957	"Deodorant Composition and Use Thereof"
5,009,887	"Deodorant Composition in the Form of a Gel"
5,693,385	"Odor Sorbing Packaging Material"

An object of the present invention is to overcome the deficiencies of the conventional paperboard based packages and containers by incorporating into the board an adsorptive material, primarily activated carbon, in a unique way, which improves the odor of the board without negatively impacting the appearance or the quality of the board.

Another object of the present invention is to provide odor improved board for all sizes and types of liquid or solid, food or non-food containers, and microwaveable and ovenable

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packaging, as required by the converter or packager to improve the market potential of the product.

Another object of this invention is to utilize this board in making liquid packaging board which may have multiple barrier layers such as aluminum foil, polyethylene terepthalate, glycol-modified PET, acid-modified PET, ethylene vinyl alcohol copolymer, polyvinyl alcohol, polybutylene terphthalate, vinylidene chloride copolymer, polyvinyl chloride polymer, vinyl chloride copolymer, polyvinyl chloride polymer, polyamide polymer, polyamide copolymer or polycarbonate polymer.

SUMMARY OF THE INVENTION

A preferred embodiment of the present invention reveals the use of an adsorptive material in the board for use in making food or non-food, liquid or solid container or package to improve inherent board odor and improve the products' market appeal.

A multi-layered paperboard composite embodying the attributes of the invention can be produced by applying granular, pelletized, fiberized, powdered, or any other form of activated carbon in between the two layers of paperboard, or on a side that will be on the inside of the package made from the paperboard, in such as way that the visual appearance and quality (*i.e.*, physical strength properties) are not negatively impacted. The paperboard which contains the activated carbon can produce various kinds of packages and containers, including paper cups and plates, which overcome the odors associated with paperboard. In addition, as a result of the method of adding this adsorbent material into the paperboard, the paperboard exhibits desirable aesthetic appearance and excellent physical characteristics. This paper/paperboard will also have

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the capability to adsorb any off-odors from printing inks and varnishes as well. Finally, the resultant package made from the invention paperboard will have the capability to adsorb any offensive odors emitted by the packaged contents.

BRIEF DESCRIPTION OF THE DRAWINGS

Figure 1 is a cross-sectional view of a multi-layer paperboard exhibiting a thin top layer and a thick bottom layer.

Figure 2 is a cross-sectional view of a multi-layer paperboard exhibiting a thin top layer, a thick bottom layer, and an adsorbent material buried within the bottom thick layer and under the top thin layer.

Figure 3 is a cross-sectional view of a single layer paperboard without added adsorbent material.

Figure 4 is a cross-sectional view of a single layer paperboard containing an added adsorbent material buried within the bottom single layer.

DESCRIPTION OF THE PREFERRED EMBODIMENT(S)

The invention is preferentially described with reference to the drawings. Figure 1 depicts a conventional multi-ply paperboard wherein the paperboard has two plies with a thin top ply (about 15% of the total board weight) and a thick bottom ply. This board normally is rated between 5-7 on taste and odor panel test. (The panel test employed in the examples to follow is described below.) Figure 2 depicts a similar board as in Figure 1, with the exception that adsorptive material has been added between the two plies in such a way that internal bonding between the two layers remains acceptable and the visual appearance of the board is not

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impaired. The adsorptive material can be applied by using various techniques such as, but not limited to, curtain coaters, conventional sprayers, air-atomized sprayers, and direct addition to the pulp stock. The adsorptive materials may be any shape of activated carbon or charcoal or equivalent thereof, as known to those skilled in the art.

Panel Test Method: A 3 gram board sample is cut into 1 square inch pieces and placed in a 1-liter wide mouth mason jar. A 100 ml beaker filled with bottled drinking water is placed into the jar without covering the board pieces. The jar is then sealed and left at ambient temperature for 24 hours. The water in the beaker is tasted by a sensory panel and compared for "degree of difference" against the control water taken directly from the bottle. The samples are rated on a scale of 1 through 7, with 1 through 3 being "acceptable," 4 being "marginal," and 5 through 7 being "unacceptable."

Application Test Method: Spray Header - A spray header made up of air atomizer nozzles was used to spray a carbon slurry at 5% solids. For a multi-ply sheet, the header was placed on the machine such that carbon slurry would fall onto the wet portion [consistency of 5-10%] of the lower sheet as it is being formed. This results in carbon getting sucked into the lower layer rather than sitting on top of the layer. A similar approach was used on a single ply.

<u>Curtain Coater</u> - A curtain coater, instead of spray header was used to apply the carbon slurry in a similar fashion described above.

Example 1

The first set of experiments was conducted using deionized (DI) water to dilute the previously dewatered secondary and primary machine chest pulps. The carbon slurry was also

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made with DI water and sprayed in between the two plies of KRAFTPAK® sheets (manufactured by Westvaco Corporation) made on the Dynamic Sheet Former (DSF). The carbon dosages tested were 0.1, 0.2, 0.5, and 1 percent (by weight). Table I shows the panel results on these samples over a five-month period. Carbon application levels of 0.5% and 1% gave excellent results throughout the five-month test period. The corresponding ratings were 2 and 3, respectively. The control was better than expected at 4, probably due to the use of DI water to dilute pulps. Thus, subsequent evaluations were done with primary and secondary head box samples without dewatering.

TABLE I

Taste and Odor Panel Results of Carbon-Treated Sheets (Pulps Diluted with DI water)

Sample ID	Carbon, %	Rating Day 1	Rating Day 4	Rating Day 7	Rating Day 11	Rating Day 105
7805-30	0	4	3	4	6	4
7805-30-4	0.1	3	2	•	4	-
7805-30-2	0.2	2	2	2	2	5
7805-30-3	0.5	2	2	2	2	2
7805-30-1	1.0	2	2	2	2	3
water blank	no paper/ carbon	1	2	2	2	1

Example 2

The same experiments were repeated using primary and secondary headbox samples.

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Since headbox samples are at about 0.5% consistency (in white water or mill process water), no additional water was required for dilution. Carbon slurries were prepared in DI water. The results are summarized in Table II.

Panel Results of Carbon-Treated Sheets (Pulps Diluted with White Water)

TABLE II.

Sample ID	Carbon %	Rating Day 1	Rating Day 7	Rating Day 14	Rating 5 Months
7805-32	0	4	6	5	6
7805-32-4	0.1	2	6	6	7
7805-32-3	0.25	2	5	5	7
7805-32-2	0.5	2	3	5	6
7805-32-1	1.0	2	3	3	3
water blank	no paper/ carbon	2	2	2	1

The day 1-panel results looked good; however, the ratings tend to revert back to poor values in subsequent testing. Only the 1% carbon level maintained good ratings for up to a five-month period. These results indicate that the white water may have a negative impact on the carbon performance at the levels tested below 1.0%. At 1.0% carbon application, results were excellent and remained so even after the 5-month period.

Example 3

Laboratory experiments were conducted to see if microporous carbon would offer better efficiency with regard to improving taste and odor. Both microporous carbon (Pica's PW-2 carbon) and HIACT® carbon (obtained from Westvaco Corporation) were tested by spraying

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aqueous slurries prepared with each type of the activated carbons, between the two KRAFTPAK® plies in the Dynamic Sheet Former. In one case, 250 ppm hydrogen peroxide was added to the primary and secondary slurries prior to sheetmaking to see if additional benefits would be gained.

Clay addition to the secondary layer was tested to enhance brightness of the sheets. These laboratory-made handsheets were panel tested nine times over a period of seven months. The results are summarized in Table III. The carbon-treated sheets with either microporous or HIACT® carbons gave and maintained acceptable taste and odor for seven months to date. The average of nine panel tests over seven months showed the (carbonless) control at an unacceptable rating of 5.2, as compared to 2.3 for HIACT® activated carbon and 2.0 for microporous activated carbon inclusions. The presence of white water in pulp slurries did not have any significant impact on panel results at the carbon levels tested. Also, the addition of hydrogen peroxide into pulp/white water slurries did not provide any clear benefit, based on panel results.

The addition of 20% Fiberex clay was found to improve the GE brightness of a carbon-treated sheet from 17.5% to a GE brightness of 20.4%.

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TABLE III

Panel Results of Carbon Treated Sheets

Exp#	Water used to make sheets	Carbon %; [on total sheet wt. basis]	Peroxide ppm, [on total slurry vol. basis]	Bright. % GE	Panel Rating Day 1	Panel Rating Day 5	Panel Rating Day 13	Panel Rating Month 1	Panel Rating Month 2	Panel Rating Month 3	Panel Rating Month 4
	Succis	Dasisj	voi. Dasisj		3/12	3/16	3/24	4/12	5/18	6/16	7/29
1. Control	ww	0	0	18.1	7	5	6	6	6	5	2
2. Control	ww	0	0	19.8 w/20% clay	6	5	6	6	6	6	4
3. Control	ww	0	250	18.1	6	4	5	6	7	6	5
4. Control	CW	0	0	18.1	5	6	5	5	7	5	5
Average					6	5	5.5	6	6.5	5.5	4
5. Hiact-1A	ww	1	0	17.5	2	2	1	3	2	2	1
6. Hiact-1B	ww	1	250	17.5	3	2	2	4	1	3	1
7. Hiact-1C	ww	1	0	20.4 w/20% clay	5	2	4	4	5	3	2
8. Hiact-1D	CW	1	0	18.0	2	2	1	2	3	2	1
Average					3	2	2	3.25	2.75	2.5	1.3
9. MP- 2A	ww	1	0	16.6	2	2	1	3	2	2	1
10. MP-2B	ww	1	250	17.0	2	2	2	2	4	2	2
11. MP-2C	CW	1	0	19.1	2	2	1	1	2	2	2
Average			,		2	2	1.3	2	2.7	2	1.7

Notes:

20% clay improved brightness of a 17.5% carbon treated sheet to 20.4%.

Peroxide added to each [primary and secondary] pulp slurry at 50 C, 10 min.

MP = Micro porous Carbon [non Westvaco]; Hiact = Westvaco Carbon

WW = White (treated mill) Water

CW = City Water

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Example 4

Paper Machine trials were run using an air atomizer and a curtain coater. These trials were successful in providing uniform carbon coverage without negatively impacting the appearance or quality of the sheet. The panel results on these mill-produced samples were found to be acceptable (in the range of 1 to 3). The carbon containing paperboard was successfully converted into prototype products. Blind panel tests on prototype products gave excellent taste and odor results as compared to the control (Table IV).

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Taste and Odor Test Results
Blind Tests in Collaboration With Sensory Directions

Table IV

Cup Type	Standard Panel Test [a]	Smell Test [b]	Taste and Odor Test w/ hot H2O [c] smell taste @ @		Taste and Odor Test w/ hot coffee [c] smell taste @ @	
Sharker in half			10 min.	20 min.	10 min.	20 min
Bleached	2 [d]	5 [e]	3	4 [e]	3	3 [e]
Kraft, Control	6	6	4	5	4	3
Kraft w/0.75% carbon	4	3	2	3	3	3
Kraft w/0.75% carbon and bleached bottom	3	2	3	3	2	4
Kraft w/1% carbon	2	2	2	3	2	3

- [a] cup clippings placed at the bottom of a jar containing a beaker full of water.

 This set up is left enclosed for 24 hours prior to tasting water for degree of difference from the control water.
- [b] cups are rated based on smell.
- [c] Test was conducted using 180°F water or coffee. Smell test was conducted at 10 minutes after hot beverage was placed into the cups [the first value in the column]. A taste test was conducted after 20 minutes [second value in the column].
- [d] Chinet bleached cups.
- [e] Commercial bleached cups from coffee shop.

Example 5

The effectiveness of a combination of activated carbon, hydrogen peroxide, and an antioxidant in improving KRAFTPAK® taste and odor was investigated. The antioxidants tested were commercially available butylated hydroxy toluene (BHT) dispersion and Oxytrap RC 91.

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Taste and odor ratings are shown in Table V. Oxytrap RC 91 showed the best results after a two-week testing.

Table V

Sample ID	Activated Carbon/ Zeolite	Type of Water	Brightness, % GE	Taste and Odor Rating	Comments
7772-62 C1	-	•	10.7	6	
7772-62 C2	-	•	12.8	7	wash primary layer with 500 ml DI water
7772-62-1	1% Carbon	ww	10.9	3 (2/3) 4 (2/17)	Sandwich
7772-62-2	1% Carbon	ww	11.2	4 (2/3) 3 (2/17)	4#/ton Oxytrap
7772-62-3	1% Carbon	ww	12.2	3 (2/3) 5 (2/17)	4#/ton BHT
7772-62-7	1% Carbon	WW neutralized with 500 ppm H ₂ O ₂	6.9	3 (2/3) 6 (2/17)	Sandwich
7772-52-1	1% Carbon	ww	-	2 (12/11) 3 (2/3) 4 (2/17)	Sandwich

WW = white water from mill. Number in parenthesis is the date sample was tested

Example 6

Different dispersing agents were used for a better carbon dispersion. KRAFTSPERSE[®] 1251, KRAFTSPERSE[®] 25M, Versa TL-70, and Ultrazine NA were used to disperse carbon. Inhouse taste and odor ratings are shown in Table VI. Ultrazine NA showed the best results. A better dispersed carbon slurry in combination with proper spray nozzle design would help further in achieving a uniform carbon application across the sheet.

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TABLE VI Impact of Carbon Dispersion Aids on Taste and Odor

ID	KRAFTSPERSE 1251	Fiberex % in Secondary	Versac TL-70	Ultrazine NA	KRAFTSPERSE 25M	Brightness %GE	Taste and Odor Rating
Control I		-			- -	14.4	4
Control II		-			-	12.2	5
Control III	•	-	•	-	-	11.3	3
Condition 1	8% (carbon weight)	-	-	-	-	11.7	3
Condition 1A	8% (carbon weight)	5				13.3	-
Condition 1B	8% (carbon weight)	10				•	-
Condition 2	•	-	8% (carbon weight)	-	-	11.8	4
Condition 3	•	•	•	8% (carbon weight)	-	10	2
Condition 4	-	-	•	•	8% (carbon weight)	12	-

Other general methods, materials, and finished products may be suggested in the instant disclosure to those skilled in the art that may differ somewhat from the specific methods, materials, and finished products reported herein. Such slight deviations are considered to be within the subject matter of this invention and within the purview of the following claims.